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Translation of selected passages from JP-A-000370/1994

Page 2, column 1, lines 1 to 46 (Claims):

【Claims】

【Claim 1】 A particulate water-absorbing agent, exhibiting a water absorption rate in the range of 20 to 90 seconds when absorbing 28 g of physiological saline per g of the water-absorbing agent, with the water-absorbing agent being characterized in that, when a steel ball of 15/32 inches in diameter is made to freely fall from the height of 20 cm onto a swollen hydrogel obtained in the above-mentioned way, the steel ball does not enter the swollen hydrogel.

【Claim 2】 A water-absorbing agent according to claim 1, of which the particle diameters are controlled by granulation to such that the ratio of particles of the size of not larger than 149 μm is less than 10 weight %, and that the ratio of particles of the size of 149 to 500 μm is not less than 50 weight %.

【Claim 3】 A water-absorbing agent according to claim 1 or 2, wherein the water absorption rate is in the range of 30 to 70 seconds.

【Claim 4】 A process for producing a water-absorbing agent, being characterized by comprising the steps of: adding an aqueous liquid containing 1 to 10 parts by weight of a polycationic compound to 100 parts by weight of crosslinked water-absorbent polymer particles; and then mixing them together; wherein the polycationic compound has a molecular weight of not less than 5,000 and is at least one member selected from the group consisting of primary, secondary, and tertiary amino groups and their salts, and wherein the crosslinked water-absorbent polymer particles have an acid group and include particles of not larger than 149 μm in particle diameter in a ratio being in the range of 15 to 75 weight %.

【Claim 5】 A process according to claim 4, wherein the ratio of particles of not larger than 149 μm in particle diameter is in the range of 25 to 65 weight %.

【Claim 6】 A process according to claim 4 or 5, wherein the polymer particles having an acid group are crosslinked polymer particles of a partially neutralized poly(acrylate salt).

【Claim 7】 A process according to claim 6, wherein the crosslinked polymer particles of the partially neutralized poly(acrylate salt) are products obtained by aqueous solution polymerization.

【Claim 8】 A process according to any one of claims 4 to 7, wherein the polycationic compound is at least one member selected from the group consisting of polyalkylenepolyamines, denatured polyethylenimines, polyallylamines, polyvinylamines and polyether amines.

【Claim 9】 A process according to any one of claims 4 to 7, wherein the polycationic compound is a polyethylenimine.

【Claim 10】 A process according to any one of claims 4 to 9, wherein the molecular weight of the polycationic compound is in the range of 10,000 to 100,000.

【Claim 11】 A water-absorbent structure, comprising a mixture of 55 to 95 parts by weight of the water-absorbing agent as recited in claims 1 to 3 and 55 to 5 parts by weight of pulverized pulp.

【Claim 12】 A body-fluid-absorbent article, comprising the water-absorbent structure as recited in claim 11.

<u>Page 4, column 6, line 41 to page 7, Table 3 bridging columns 11 and 12 (Examples and Comparative Examples)</u>:

【0023】

【Working Examples】 Hereinafter, the present invention is more specifically illustrated by the following examples of some preferred embodiments, but is not limited thereto.

【0024】 Referential Example 1: Synthesis of Water-Absorbent Resin Particles (A):

Polymerization of 4,000 weight parts of 39 % aqueous solution of acrylate

salt-based monomers, comprising sodium acrylate 74.95 mol %, acrylic acid 25 mol %, and trimethylolpropane triacrylate 0.05 mol %, was carried out with 5.0 weight parts of sodium persulfate and 0.25 weight part of L-ascorbic acid at 30-70 °C under a nitrogen atmosphere to obtain a crosslinked hydrogel copolymer. The resultant hydrogel polymer was dried with a hot-air drying machine of 150 °C, and then pulverized with a hammer mill, and then sieved with a metal gauze of the mesh opening size of 500 μm (32-mesh standard sieve according to JIS), thus obtaining a 32-mesh-passed product (hereinafter referred to as water-absorbent resin particles (A)). Its particle diameter distribution was as follows: particles of 149-500 μm = 68.7 weight %, particles of not larger than 149 μm = 31.3 weight %.

【0025】 Referential Example 2: Synthetic Example of Water-Absorbent Resin Particles (B):

Polymerization of 4,000 weight parts of 20 % aqueous solution of acrylic acid-based monomers, comprising acrylic acid 99.8 mol % and methylenebisacrylamide 0.2 mol %, was carried out with 12 weight parts of azo type initiator V-50 (produced by Wako Pure Chemical Industries, Ltd.), 4 weight parts of hydrogen peroxide, and 1 weight part of L-ascorbic acid at 10-80 °C under a nitrogen atmosphere to obtain a crosslinked hydrogel polymer. The resultant hydrogel polymer was disintegrated, and then thereto 2,224 weight parts of sodium hydroxide was added to mix them together. Thereafter, the resultant mixture was dried with a hot-air drying machine of 150 °C, and then pulverized with a hammer mill, and then sieved with a metal gauze of the mesh opening size of 500 μm, thus obtaining water-absorbent resin particles (B) not larger than 500 μm. Their particle diameter distribution was as follows: particles of 149-500 μm = 30 %, particles of not larger than 149 μm = 70 %.

【0026】 Example 1:

An amount of 6 weight parts of Epomin P-1050 (50 % aqueous solution of polyethylenimine having a number-average molecular weight of about 70,000; produced

by Nippon Shokubai Co., Ltd.) was added to 100 weight parts of the water-absorbent resin particles (A) to mix them together, and then the resultant mixture was left alone at room temperature for 1 hour, and then disintegrated. All the resultant particles were passed through a metal gauze of the mesh opening size of 840 μm, and the resultant product was further mixed with 0. 5 weight part of Aerosil 200 (superfine particulate silicon oxide, produced by Nippon Aerosil Co., Ltd.), thus obtaining a water-absorbing agent (1) according to the present invention. Its particle diameter distribution was as follows: particles of 149-500 μm = 85.4 weight %, particles of not larger than 149 μm = 2.2 weight %.

【0027】 Example 2:

The water-absorbent resin particles (A) were further sieved with a metal gauze of the mesh opening size of 297 μm (48-mesh standard sieve according to JIS), thus obtaining a 48-mesh-passed product (hereinafter referred to as water-absorbent resin particles (C)). Its particle diameter distribution was as follows: particles of not larger than 149 μm = 46.2 weight %. An aqueous liquid, comprising 6 weight parts of Epomin P-1050 (50 % aqueous solution of polyethylenimine having a number-average molecular weight of about 70,000; produced by Nippon Shokubai Co., Ltd.) and 2 weight parts of ethanol, was added to 100 weight parts of the water-absorbent resin particles (C) to mix them together, and then the resultant mixture was left alone at 70 °C for 15 minutes, and then disintegrated. All the resultant particles were passed through a metal gauze of the mesh opening size of 840 μm, and the resultant product was further mixed with 0. 5 weight part of Aerosil 200 (superfine particulate silicon oxide, produced by Nippon Aerosil Co., Ltd.), thus obtaining a water-absorbing agent (2) according to the present invention. Its particle diameter distribution was as follows: particles of 149-500 μm = 90.3 weight %, particles of not larger than 149 μm = 2.7 weight %.

【0028】 Example 3:

An aqueous liquid, comprising 6 weight parts of Epomin P-1050 (50 % aqueous solution of polyethylenimine having a number-average molecular weight of about 70,000; produced by Nippon Shokubai Co., Ltd.) and 2 weight parts of ethanol, was added to 100 weight parts of the water-absorbent resin particles (B) to mix them together, and then the resultant mixture was left alone at room temperature for 1 hour. All the resultant particles were passed through a metal gauze of the mesh opening size of 840 μm, and the resultant product was further mixed with 0. 5 weight part of Aerosil 200 (superfine particulate silicon oxide, produced by Nippon Aerosil Co., Ltd.), thus obtaining a water-absorbing agent (3) according to the present invention. Its particle diameter distribution was as follows: particles of 149-500 μm = 76 weight %, particles of not larger than 149 μm = 4.7 weight %.

【0029】 Comparative Example 1:

The water-absorbent resin particles (A) were further sieved with a metal gauze of the mesh opening size of 149 μm (100-mesh standard sieve according to JIS), thus obtaining a 100-mesh-passed product (hereinafter referred to as water-absorbent resin particles (D)). An amount of 100 weight parts of the water-absorbent resin particles (D) were treated in the same way as of Example 2, thus obtaining a comparative water-absorbing agent (1). Its particle diameter distribution was as follows: particles of 149-500 μm = 62.8 weight %, particles of not larger than 149 μm = 19.0 weight %.

【0030】 Comparative Example 2:

An amount of 5 weight parts of 10 % aqueous solution of aluminum sulfate was added to 100 weight parts of the water-absorbent resin particles (A) to mix them together, and then the resultant mixture was left alone at room temperature for 40 minutes and then mixed with 1 weight part of Aerosil 200 (superfine particulate silicon oxide, produced by Nippon Aerosil Co., Ltd.). The resultant mixture was passed through a sieve of the mesh opening size of 840 μm, thus obtaining a comparative water-absorbing agent (2). Its particle diameter distribution was as follows: particles

of 149-500 μm = 67.7 weight %, particles of not larger than 149 μm = 5.5 weight %.

【0031】 Examples 4 to 6 and Comparative Examples 3 to 4:

The water-absorbing agents (1) to (3) and the comparative water-absorbing agents (1) to (2), as obtained in Examples 1 to 3 and Comparative Examples 1 to 2, were evaluated in the following ways. The results are shown in Table 1.

【0032】(Water absorption rate): Into a cylindrical polypropylene cup of 50 mm in inner diameter and 70 mm in height, 1 g of water-absorbing agent was placed and then 28 g of physiological saline was poured to uniformly be absorbed. The time of from the beginning of the above pouring till gelation of the entire physiological saline (state where the physiological saline was unseen throughout the surface) was measured, and the average of three times of such measurement was defined as the value.

【0033】 (Ball-falling test): After the measurement of the water absorption rate, the gel as swollen to 28 g/g in the cup was left alone for 10 minutes. Thereafter, a steel ball (diameter = 15/32 inches, weight = 6.9 g: a steel ball according to JIS B-1501) was made to freely fall from the height of 20 cm onto the resultant swollen hydrogel. The distance by which the ball bounded from or entered the swollen hydrogel was measured. Such a test was made three times, and its average was determined.

【0034】 Example 7:

An amount of 140 weight parts of water-absorbing agent (1), as obtained in Example 1, and 60 weight parts of pulverized pulp were mixed together in a mixer in a dry manner. Next, the resultant mixture was pneumatically molded on a wire screen with a batch type pneumatic molding device to form a web of the measurements of 10 cm × 20 cm. The top and bottom of the resultant web were sandwiched between tissues of the basis weight of 0.0013 g/cm^2, and then they were pressed under a pressure of 2 kg/cm^2 for 5 seconds to obtain a water-absorbent structure (1) according to the present invention having a basis weight of about 0.05 g/cm^2 and a density of about 0.17 g/cm^3.

【0035】 Examples 8 to 9 and Comparative Examples 5 to 6:

The water-absorbing agents (2) to (3), as obtained in Examples 2 to 3, and the comparative water-absorbing agents (1) to (2), as obtained in Comparative Examples 1 to 2, were used to obtain water-absorbent structures (2) to (3) according to the present invention and comparative water-absorbent structures (1) to (2) in the same way as of Example 7.

【0036】 Examples 10 to 12 and Comparative Examples 7 to 8:

The water-absorbent structures (1) to (3) according to the present invention and the comparative water-absorbent structures (1) to (2), as obtained above, were evaluated in the following ways, thereby evaluating the water absorption properties of the water-absorbent structures. The results are shown in Table 1.

【0037】 (Evaluation of Water-Absorbent Structure Without Load): The resultant water-absorbent structure was cut into the shape of a circle of 9 cm in diameter and then placed into a Petri dish of 120 mm in diameter and 40 mm in height, and then 50 g of physiological saline was poured thereinto. The time of from the beginning of the above pouring till absorption of the entire physiological saline into the water-absorbent structure was measured, and the average of three times of such measurement was defined as the value.

【0038】 (Evaluation of Water-Absorbent Structure Under Load): The resultant water-absorbent structure was cut into the shape of a circle of 9 cm in diameter and then placed into a Petri dish of 120 mm in diameter and 40 mm in height, and then 30 g of physiological saline was poured thereinto. The time of from the beginning of the above pouring till absorption of the entire physiological saline into the water-absorbent structure was measured (without load). After being left alone for 5 minutes, a load of 12 g/cm^2 was applied to the entire water-absorbent structure, and in that state, 20 g of physiological saline was further poured thereon. The time of from the beginning of the above pouring till absorption of the entire physiological saline into the water-absorbent

structure was measured (under load). The average of three times of such measurement was defined as the values.

【0039】

【Table 1】

	Evaluation as water-absorbing agent		Evaluation as water-absorbent structure	
	Water absorption rate (sec)	Entering distance (mm)	Without load (sec)	Without load/under load (sec)
Water-absorbing agent (1)	60	0	25	8/10
(2)	45	0	22	9/6
(3)	31	0	17	9/9
Comparative water-absorbing agent (1)	19	0	13	5/40
(2)	36	>14	23	6/21

【0040】 Example 13:

An amount of 150 weight parts of water-absorbing agent (1), as obtained in Example 1, and 150 weight parts of pulverized pulp were mixed together in a mixer in a dry manner. Next, the resultant mixture was pneumatically molded on a wire screen with a batch type pneumatic molding device to form a web of the measurements of 10 cm × 20 cm. The top and bottom of the resultant web were sandwiched between tissues of the basis weight of 0.0013 g/cm^2, and then they were pressed under a pressure of 2 kg/cm^2 for 5 seconds to obtain a water-absorbent structure (4) according to the present invention having a basis weight of about 0.05 g/cm^2 and a density of about 0.17 g/cm^3.

【0041】 Examples 14 to 15 and Comparative Examples 9 to 10:

The water-absorbing agents (2) to (3), as obtained in Examples 2 to 3, and the comparative water-absorbing agents (1) to (2), as obtained in Comparative Examples 1 to 2, were used to obtain water-absorbent structures (5) to (6) according to the present invention and comparative water-absorbent structures (3) to (4) in the same way as of Example 13.

【0042】 Examples 16 to 21 and Comparative Examples 11 to 14:

The water-absorbent structures (1) to (6) according to the present invention and the comparative water-absorbent structures (1) to (4), as obtained above, were evaluated in the following ways, thereby evaluating the water absorption properties of the water-absorbent structures. The results are shown in Table 2.

【0043】 (Evaluation of Primary and Secondary Absorptions of Water-Absorbent Structure Under Load): The resultant water-absorbent structure was cut into the shape of a circle of 9 cm in diameter and then placed into a Petri dish of 120 mm in diameter and 40 mm in height. On the water-absorbent structure there was mounted an acrylic resin plate of 9 cm in diameter, and further thereon a weight was put, thus applying a load of 14 g/cm^2 to the entire water-absorbent structure.

【0044】 In that state, 25 g of physiological saline was poured to measure the time of from the beginning of this pouring till absorption of the entire physiological saline into the water-absorbent structure was measured (primary absorption). After 5 minutes, 25 g of physiological saline was further poured to measure the time of till re-absorption of the entire physiological saline into the water-absorbent structure (secondary absorption).

【0045】

【Table 2】

	Water absorption rate under load	
	Primary absorption (sec)	Secondary absorption (sec)
Water-absorbent structure (1)	36	57
(2)	27	55
(3)	24	56
(4)	35	91
(5)	27	64
(6)	23	56
Comparative water-absorbent structure (1)	25	138
(2)	29	109
(3)	19	156
(4)	29	108

【0046】 Example 22:

An amount of 120 weight parts of water-absorbing agent (1), as obtained in Example 1, and 80 weight parts of pulverized pulp were mixed together in a mixer in a dry manner. Next, the resultant mixture was pneumatically molded on a wire screen with a batch type pneumatic molding device to form a web of the measurements of 15 cm × 40 cm. The top and bottom of the resultant web were sandwiched between tissues of the basis weight of 0.0013 g/cm^2, and then they were pressed under a pressure of 2 kg/cm^2 for 5 seconds to obtain a water-absorbent structure (7) according to the present invention having a basis weight of about 0.05 g/cm^2 and a density of about 0.17 g/cm^3 (weight = 24 g).

【0047】 An absorbent article (1) according to the present invention, comprising a liquid-permeable polypropylene top sheet, the water-absorbent structure (7) according to the present invention, a liquid-impermeable polyethylene back sheet (including leg gathers), and two tape fasteners, was hand-assembled by binding these components to each other with a double-coated tape. The weight of the absorbent article (1) according to the present invention was 45 g.

【0048】 Comparative Example 15:

The comparative water-absorbing agent (2), as obtained in Comparative Example 2, was used to produce a comparative absorbent article (1) in the same way as of Example 22. The weight of the comparative absorbent article (1) was 45 g.

【0049】 Example 23:

The absorbent article (1) according to the present invention and the comparative absorbent article (1) were tested by eight mother panelists (babies aged 1) through about 1 month. Each panelist received twenty diapers at random to make the test. After the test, the diapers were collected to compare their leakage ratios. The results are shown in Table 3. The absorbent article according to the present invention is found to have excellent absorption properties.

【0050】

【Table 3】

	Absorbent article (1)	Comparative absorbent article (1)
Total number of diapers	80	80
Number of times of leakage	5	12
Leakage ratio (%)	6.3	15